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### Novel Synthesized Disperse Dyes based on Enaminones Provide Added-Value: Part 1



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#### Abstract

In this review article paper, we present the synthesis of a series of disperse — dyes based on enaminones, and we explain precisely that they provided an added value, not only as their use in dyeing polyester fabrics, but also because these dyes have activity against some bacteria and cancer.

Keywords: Disperse dyes, .Low and High temperature dyeing methods, Antibacterial activities

#### 1. Introduction

It should be emphasised that due to enaminone's success in creating various chemical compounds, its application has increased during the past ten years [1-45]. Enaminones are recognised as polydentate reactants with significant roles in organic chemistry [6-14]. Enaminones have been used as intermediates in the design of various naturally dynamic chemicals [15-32]. Enaminones have been used as precursors to polyfunctional organic chemistry in previous studies [33-44]. In our presentation hown below, we presented the effectiveness of new azo disperse dyes made from enaminones that may be used to dye polyester garments using a variety of dyeing methods [1-9]. There is interest in polymers with ketoester side groups that can be created by dynamic enaminone connections and the development of postfunctionalization techniques of high-performance components, including ceramics, coatings, and dentistry resins [51].

#### 2. Characterizations

According to Scheme 1, methylketones Ia-c were condensed with dimethylformamide and dimethyl acetal to produce enaminones IIa-c in

respectable yields. The coupling reaction between enaminone IIa-c and arylidene diazonium chloride was the next thing we looked at. This process controlled **1-6** novel azo disperse dyes.

## 3. Low Temperature Dyeing of Polyester Fabrics

### 3.1. Effect of carrier on K/S

According to the findings in Table 1, the colour strength K/S values of polyester textiles dyed with colours 1, 2, and 5 disperse dyes rise with increasing carrier concentration and reach their highest value (10.08, 11.24, and 10.13) at 1% concentration. After that, they start to decline. The colour strength K/S values of polyester fabrics coloured with disperse dyes 3 and 6 rise to their highest value (6.66 and 10.10) at 0.5% concentration before falling with increasing carrier concentration. Finally, at 2% carrier concentration, the K/S values of polyester fabrics treated with dye No. 4 reach their highest value (14.04).

### 3.2. Effect of dispersing agent on K/S

According to the findings in Table 2, the K/S values of the polyester textiles coloured with the

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disperse dyes of dyes 1, 3, 5, and 6 rise with an increase in the concentration of the dispersing agent and reach their highest value (10.85, 10.67, 13.01, and 7.54) at a concentration of 1.5%. With increasing concentrations of the dispersing agent, the K/S values for the polyester fabric coloured with the disperse dyes of the 2 and 4 colours likewise rise, reaching their highest values (10.23 and 10.36) at a concentration of 2%Table 2: Dispersing agent effects on the dyeing process of disperse dyes.

# 3.3. Relation between dye concentrations of the disperse dyes and K/S

The colour strength K/S values for polyester textiles coloured with dispersion dyes of colours 1, 2, 3, 5 and 6 rise with increasing dye concentration and reach their highest value (21.91, 15.08, 17.43, 19.37, and 3.40) at dye concentration 2%, according to Table 3 and Figure 1. For the polyester fabric coloured with disperse dyes No. 4, the colour strength K/S values rise with increasing dye concentration and reach their highest value (13.75) at the dye concentration of 3%.

#### 3.4. Fastness properties

The results are shown in Tables 4-6, and they demonstrate that the fastness against washing offered very good results, with the shade of 2% outperforming both shades 1% and 3%. The fastness qualities of polyester fabrics coloured with the novel dispersion dyes have been done at shades from 1% to 3%.

# 4. High Temperature Dyeing of Polyester Fabrics

#### 4.1. Effect of dispersing agent on K/S

The results shown in Table 7 make it abundantly evident that 1.5% dispersing agent is the ideal concentration to use for dying polyester fabrics with fresh dispersed colours at a temperature of 130 °C.

# 4.2. Relation between dye concentrations of the disperse dyes and K/S

The colour strength K/S values for polyester textiles coloured with disperse dyes of colours 1- 6 grow with increasing dye concentration and reach their highest value (17.14, 15.54, 13.76, 19.93, 16.47, and 13.95) at dye concentration 3%, according to Table 8 and Figure 2.

#### 4.3. Fastness properties

The results are shown in Tables 9–11, and they demonstrate that the fastness against washing, rubbing, and perspiration offered very good results. The fastness properties of polyester fabrics dyed with the novel disperse dyes have been done at shades from 1% to 3%. The findings of the fastness properties against light were satisfactory; shade 1% performed somewhat better than shades 2% and 3%, and shade 2% performed slightly better than shade 3%.

#### 5. Dyebath Reuse and Biological Activities

#### 5.1. Dyebath reuse.

According to the data from table (12), which is depicted in figure (3), the colour strength measurement K/S value of the dyebath reuse process in the dyeing process at 100 °C varies by 10, 20, 30, 60, and 90% of its initial value in the first dyeing process. This indicates that dyeing reuse is an efficient way to cut costs, prevent pollution, and save on chemicals, water, energy, and other resources.

From the data found in table (13), which is depicted in figure (4), we deduced that the colour strength measurement K/S value of the dyebath reuse process in the dyeing process at 130 °C equals approximately 5–10% of its original value in the first dyeing process. This proves that HTPM is a preferred dyeing method because it provides a good colorful shade. It also demonstrates that dyeing reuse is an efficient method of lowering costs, pollution prevention, water, energy.

#### 5.2. Antimicrobial activities.

Against gramme positive, gramme negative, and fungal bacteria and fungi, dye 1 exhibits robust activity with an inhibition zone > 9 mm. While Dye 2 exhibits no activity against Aspergillus flavus, it exhibits high activities with an inhibitory zone > 9 mm against gramme positive, gramme negative, and candida albicans. In contrast to aspergillus flavus and candida albicans, dye 3 has robust activity with a considerable inhibition zone >9 mm against gramme positive and negative microorganisms. Against every studied bacterium, dye 4 exhibits high activation with a considerable inhibition zone larger than 11 mm. Against every investigated bacterium, dye 5 exhibits robust activation with a considerable inhibition zone

larger than 10 mm. In contrast to Aspergillus flavus, Dye 6 has robust activities with a considerable inhibition zone >9 mm against gramme positive, negative microorganisms and candida albicans.

**Table 1:** Carrier effects on the dyeing process of disperse dyes.

Dye	% carrier	L*	<u>a*</u>	<i>b</i> *	K/S
No	, v vui 1101	_		v	12/2
	0.5%	81.35	-10.33	53.63	9.07
	1.0%	81.24	-10.18	56.44	10.08
1	1.5%	81.54	-10.42	55.70	9.82
	2.0%	81.38	-9.94	57.43	8.70
	0.5%	81.67	-11.52	54.20	10.51
	1.0%	81.49	-11.41	54.85	11.24
2	1.5%	82.25	-12.81	52.10	10.19
	2.0%	82.79	-13.29	53.19	10.43
	0.5%	83.18	-12.91	44.47	6.66
	1.0%	83.02	-12.75	45.50	6.53
3	1.5%	82.96	-12.30	45.54	6.22
	2.0%	82.97	-12.25	45.82	6.40
	0.5%	80.33	-5.64	40.41	12.95
	1.0%	81.35	-6.52	38.62	10.08
4	1.5%	80.62	-6.62	38.87	11.48
	2.0%	81.09	-6.33	40.58	14.04
	0.5%	81.28	-6.06	37.91	8.48
	1.0%	81.61	-6.33	36.91	10.13
5	1.5%	82.11	-7.04	36.08	7.94
	2.0%	81.13	-6.07	38.83	8.81
	0.5%	81.79	-6.92	37.76	10.10
6	1.0%	81.32	-6.75	39.03	7.24
	1.5%	81.32	-6.63	39.63	6.96
	2.0%	81.38	-7.26	38.60	7.41

CH<sub>3</sub>

$$X = H, \\ b, X = CH_3, \\ c, X = Br$$

$$X = H_3C$$

$$X = H_4$$

$$B_1 = X = H, \\ b, X = CH_3, \\ c, X = Br$$

$$X = R_1$$

$$A_2 = R_2$$

$$A_3 = R_3$$

$$A_4 = R_4$$

$$A_4 = R_4$$

$$A_5 = R_4$$

$$A_5 = R_4$$

$$A_5 = R_5$$

$$A_6 = R_5$$

$$A_7 = R_7$$

$$A_7 = R_7$$

$$A_8 = R_7$$

$$A_9 = R_7$$

**Scheme 1**: Synthesis of azo disperse dyes 1-6.

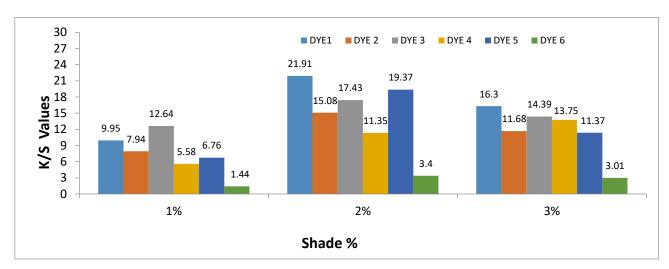


Figure 1. Relation between dye concentrations and shades of the disperse dyes.

**Table 2:** Dispersing agent effects on the dyeing process of disperse dyes.

Dye	% Dispersing	L*	a*	<i>b</i> *	K/S
No	agent				
	0.5%	81.14	-10.13	57.18	9.46
	1%	81.77	-10.41	57.08	9.78
1	1.5%	80.78	-10.39	57.13	10.85
	2%	80.99	-10.37	57.26	10.36
	0.5%	82.24	-13.61	51.69	9.41
	1%	82.20	-13.54	50.77	9.89
2	1.5%	82.75	-13.10	50.97	8.48
	2%	80.08	-12.98	50.44	10.23
	0.5%	81.71	-12.30	54.77	9.29
	1%	82.74	-13.64	51.44	9.31
3	1.5%	82.43	-13.01	51.32	10.67
	2%	82.13	-13.11	51.89	10.24
	0.5%	80.82	-6.12	38.11	9.37
	1%	81.03	-5.87	36.92	7.88
4	1.5%	80.69	-6.33	37.43	7.15
	2%	81.36	-6.54	36.42	10.36
	0.5%	81.06	-6.67	36.84	8,64
	1%	82.17	-7.01	36.80	9.61
5	1.5%	81.95	-6.82	35.82	13.01
	2%	81.50	-6.64	38.91	9.89
	0.5%	82.23	-6.72	33.42	4.44
6	1%	82.30	-7.26	33.73	6.62
	1.5%	82.05	-6.91	34.70	7.54
	2%	81.76	-7.32	37.22	7.29

**Table 3:** Effect of the dye shades used in dyeing process at 100 °C and K/S of dyed fabrics.

Dye No	% shade	L*	a*	<i>b</i> *	K/S
	1%	87.88	-5.65	67.75	9.95
1	2%	85.63	-2.60	81.84	21.91
	3%	83.94	-1.03	83.13	16.30
	1%	87.44	-9.58	60.92	7.94
2	2%	88.07	-8.19	71.01	15.08
	3%	86.45	-8.60	67.57	11.68
	1%	88.46	-9.35	68.29	12.64
3	2%	85.09	-6.96	71.83	17.43
	3%	87.12	-3.89	78.98	14.39
	1%	87.68	-1.11	47.54	5.58
4	2%	83.19	-1.20	53.54	11.35
	3%	81.17	-0.23	51.38	13.75
	1%	83.22	-3.31	45.18	6.76
5	2%	75.14	-1.59	48.83	19.37
	3%	83.62	-1.38	50.11	11.37
	1%	88.59	-4.68	26.43	1.44
6	2%	86.77	-3.98	36.28	3.40
	3%	85.30	-2.29	37.78	3.01

**Table 4:** Fastness properties of disperse dyes on polyester fabrics at shade 1%

Dye		Washing	<u> </u>	Rubbing Perspiration fastness								
No		fastness	ness fastness				Acidic			Alkaline		Light
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	fastness
1	4	4	4	4-5	4-5	4-5	4	4	4-5	4-5	4	3
2	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	3
3	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
4	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	3
5	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4-5	4	3
6	4-5	4	4-5	4	4-5	4	4	4	4-5	4	4	2-3

**Table 5:** Fastness properties of disperse dyes on polyester fabrics at shade 2%

Dye		Washing		Rub	bing			Perspira	tion fastr	iess			
No		fastness		fast	ness		Acidic Alkaline					Light	
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	fastness	
1	4-5	4	4-5	4-5	4	4-5	4-5	4	4-5	4-5	4	2-3	
2	4-5	4-5	4-5	4-5	4	4-5	4	4	4-5	4	4	3	
3	4-5	4	4-5	4	4	4	4	4	4-5	4-5	4	2-3	
4	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3	
5	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3	
6	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	3-4	

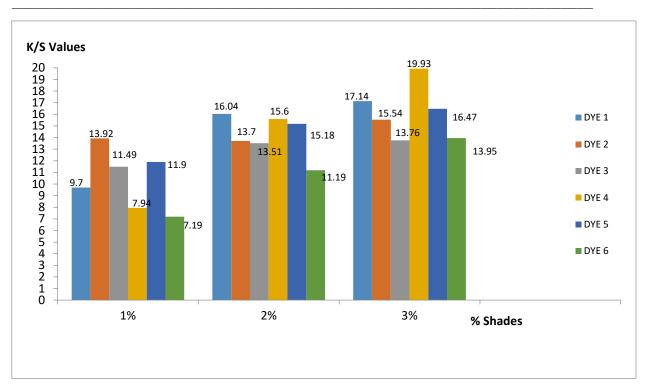


Figure 2. Relation between dye concentrations and shades

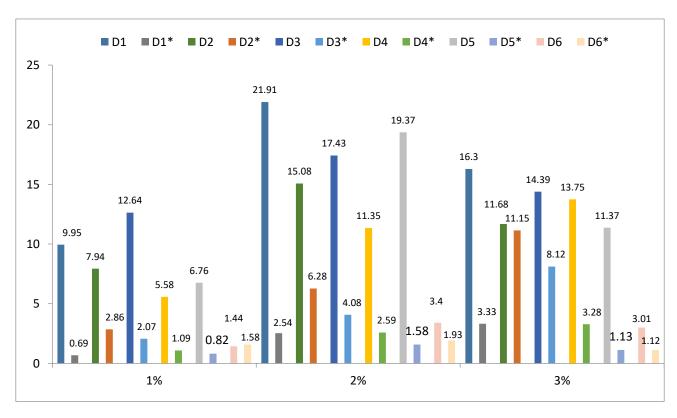


Figure 3. Effect of the dyebath reuses dyeing process at 100 °C and K/S of dyed fabrics

**Table 6:** Fastness properties of disperse dyes on polyester fabrics at shade 3%

Dye No		Washing fastness	,		bing ness			Light				
110		Tastifess	•	last	ness	Acidic			Alkaline			fastness
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	-
1	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
2	4-5	4	4-5	4-5	4	4-5	4-5	4	4-5	4	4	2–3
3	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4	4	2-3
4	4-5	4	4-5	4	4	4	4	4	4-5	4-5	4	2-3
5	4-5	4	4-5	4-5	4	4-5	4-5	4	4-5	4	4	2–3
6	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4-5	4	3–4

**Table 7:** Dispersing agent effects on the dyeing process of disperse dyes.

Dye No	% Dispersing agent	L*	a*	<i>b</i> *	K/S
	0.5%	83.08	-11.19	57.60	11.80
	1%	80.30	-9.12	57.25	9.94
1	1.5%	81.63	-10.29	57.58	13.18
	2%	80.70	-10.17	57.31	10.88
	0.5%	81.19	-11.61	56.42	12.34
	1%	81.85	-12.04	56.33	16.99
2	1.5%	82.78	-12.12	56.60	11.63
	2%	81.76	-12.43	56.92	15.85
	0.5%	82.03	-12.19	52.52	9.43
	1%	81.68	-12.20	53.78	10.96
3	1.5%	82.79	-12.75	51.75	10.31
	2%	81.66	-11.10	53.72	10.60
	0.5%	78.40	-5.11	33.44	7.48
	1%	77.95	-5,90	37.40	9.53
4	1.5%	81.64	-7.19	38.56	10.27
	2%	80.60	-6.22	39.57	9.47
	0.5%	80.63	-5.85	38.02	11.13
	1%	82.47	-6.94	35.54	9.03
5	1.5%	80.32	-6.59	37.88	12.41
	2%	79.19	-5.82	34.30	8.57
	0.5%	81.98	-7.54	38.20	10.60
6	1%	81.65	-6.51	36.28	4.67
	1.5%	81.14	-6.54	39.58	9.22
	2%	81.78	-7.74	35.58	7.48

**Table 8:** Effect of the dye shades used in dyeing process at 100 °C and K/S of dyed fabrics.

Dye No	% shade	L*	a*	<i>b</i> *	K/S
	1%	86.92	-7.92	65.08	9.70
1	2%	84.86	-3.70	79.77	16.04
	3%	83.26	-0.12	83.09	17.14
	1%	88.18	-12.01	64.81	13.92
2	2%	86.89	-9.44	70.33	13.70
	3%	85.47	-7.64	75.87	15.54
	1%	88.83	-13.10	62.39	11.49
3	2%	89.66	-9.77	73.76	13.51
	3%	86.63	-9.02	75.02	13.76
	1%	86.78	-4.95	42.07	7.94
4	2%	82.00	1.29	53.86	15.60
	3%	78.44	5.33	59.76	19.93
	1%	84.60	-1.77	48.85	11.90
5	2%	82.48	0.92	54.46	15.18
	3%	81.06	2.85	57.38	16.47
	1%	87.53	-6.23	41.10	7.19
6	2%	85.33	-3.24	48.30	11.19
	3%	79.00	-3.11	47.46	13.95

 Table 9: Fastness properties of disperse dyes on polyester fabrics at shade 1%

Dye		Washing		Rub	bing			Perspira	tion fastr	iess		
No	o fastness			fast	ness	Acidic			Alkaline			Light
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	fastness
1	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4	4	3
2	4-5	4	4	4-5	4	4-5	4	4	4-5	4-5	4	3
3	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4-5	4	3
4	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	4
5	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	3
6	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	3-4

**Table 10:** Fastness properties of disperse dyes on polyester fabrics at shade 2%

Dye		Washing		Rub	bing			Perspira	tion fastn	ess		
No		fastness	fast	fastness		Acidic			Alkaline		Light	
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	- fastness
1	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	2-3
2	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	3
3	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4	4	2-3
4	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4-5	4	3-4
5	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	3
6	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4-5	4	3

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**Table 11:** Fastness properties of disperse dyes on polyester fabrics at shade 3%

Dye		Washing		Rub	bing			Perspira	tion fastr	ess		
No	To fastness			fast	ness		Acidic			Alkaline		
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	fastness
1	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	2-3
2	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4-5	4	2-3
3	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4	4	2-3
4	4-5	4	4	4-5	4	4-5	4-5	4	4-5	4	4	3-4
5	4-5	4	4	4-5	4	4-5	4	4	4-5	4	4	2-3
6	4-5	4	4	4-5	4	4-5	4	4	4-5	4-5	4	3

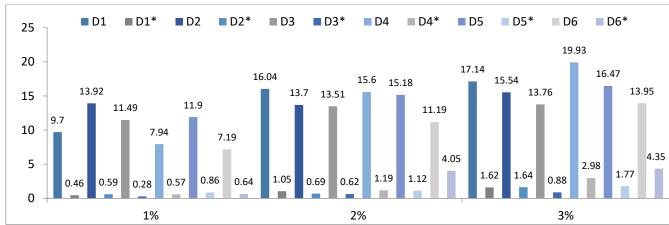


Figure 4. Effect of the dyebath reuses dyeing process at 100 0C and K/S of dyed fabrics

Table 12: Effect of the dye bath reuses dyeing process at 100 0C and K/S of dyed fabrics

Dye No.	% shade				K	/S
·		$L^*$	$a^*$	$b^*$	Dye	First
					reused	dyeing
	1%	91.30	-5.52	23.47	0.69	9.95
1	2%	91.17	-8.10	46.30	2.54	21.91
	3%	90.03	-8.22	50.88	3.33	16.30
	1%	91.95	-9.97	46.55	2.86	7.94
2	2%	88.36	-11.27	52.64	6.28	15.08
	3%	87.06	-10.15	63.06	11.15	11.68
	1%	89.03	-9.89	38.23	2.07	12.64
3	2%	88.09	-10.54	50.11	4.08	17.43
	3%	87.38	-10.85	58.48	8.12	14.39
	1%	89.38	-4.94	20.16	1.09	5.58
4	2%	89.69	-3.87	35.79	2.59	11.35
	3%	86.90	-4.80	35.11	3.28	13.75
	1%	92.15	-4.07	19.65	0.82	6.76
5	2%	88.75	-5.27	24.02	1.58	19.37
	3%	84.50	-4.33	48.04	1.13	11.37
	1%	89.65	-6.24	23.03	1.58	1.44
6	2%	88.72	-5.96	26.84	1.93	3.40
	3%	87.49	-3.18	22.64	1.12	3.01

**Table 13:** Effect of the dye bath reuses dyeing process at  $130~^{0}$ C and K/S of dyed fabrics

Dye	% shade				K	/S
No		$L^*$	$a^*$	$b^*$	Dye	First
					reused	dyeing
	1%	90.43	-5.32	17.41	0.46	9.70
1	2%	89.92	-7.67	29.48	1.05	16.04
	3%	88.97	-7.80	38.29	1.62	17.14
	1%	90.64	-5.86	17.77	0.59	13.92
2	2%	90.61	-6.58	20.43	0.69	13.70
	3%	90.40	-9.42	32.72	1.64	15.54
	1%	91.41	-4.19	11.12	0.28	11.49
3	2%	90.80	-6.72	19.80	0.62	13.51
	3%	90.44	-7.87	24.61	0.88	13.76
	1%	90.59	-3.81	12.04	0.57	7.94
4	2%	92.27	-4.38	22.65	1.19	15.60
	3%	89.03	-6.21	29.39	2.98	19.93
	1%	90.79	-5.00	15.66	0.86	11.90
5	2%	90.44	-5.51	18.42	1.12	15.18
	3%	98.94	-5.91	24.01	1.77	16.47
•	1%	91.51	-4.67	12.28	0.64	7.19
6	2%	89.65	-8.34	31.64	4.05	11.19
	3%	89.98	-6.32	37.63	4.35	13.95

Table 14. Antitumor and antioxidant activities of the disperse dyes 1-6

Dye	Cytotoxic activity (IC <sub>50</sub> µg/ml)	Antioxidant activity
Number	HepG-2	$(IC_{50} \mu g/ml)$
Disperse dye 1	$222 \pm 6.20$	63.80
Disperse dye 2	$0.96 \pm 0.08$	111.0
Disperse dye 3	$1.66 \pm 0.12$	74.90
Disperse dye 4	> 500	158.7
Disperse dye 5	$1.96 \pm 0.18$	153.4
Disperse dye 6	$13.0 \pm 1.20$	150.5
Doxorubicin	0.42	
Imatinib	18.9	
Ascorbic acid		14.20

#### 5.3. Cytotoxicity activities.

According to the information in table (14), the descending order of the tested compounds' in vitro inhibitory effects against the hepatocellular

carcinoma cell line (HEP G2) is as follows: D2>D3>D5>D6>D1.D4.

#### 5.4. Antioxidant activities.

According to the information found in table (4), the examined compounds' in vitro antioxidant activity are organised in the following descending order: D1>D3>D2>D6>D5>D4. Ascorbic acid, a well-known antioxidant that scavenges the DPPH radical, has an IC50 value of 14.2 g/ml. Additionally, it appears that these dyes' ability to scavenge free radicals is primarily due to their azo groups, which exhibit strong antioxidant activity similar to that of phenolic compounds. These compounds are distinguished by their capacity to act as donating groups, in addition to the position and type of the sustituent that is bonded to the aromatic ring, which

may cause a slight increase or decrease in antioxidant activity.

#### 6. Conclusions

From the above, it has become clear beyond doubt that the dispersed dyes that we have synthesized have an added value because, in addition to their use in dyeing polyester fabrics, they have an excellent and promising biological activity that qualifies them for possible use in pharmaceutical and medicinal chemistry.

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